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Modification of mass transfer properties in poplar wood (*P. robusta*) by a thermal treatment at high temperature

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Abstract This paper is devoted to the measurement and analysis of the mass transfer properties in the hygroscopic range of wood specimens of poplar (*Populus robusta*) before and after a treatment at 200°C. In this work, two different properties have been determined on the same samples: the mass diffusivity (property of mass migration under the effect of a concentration gradient) and the air permeability (property of mass migration under the effect of a pressure gradient). These properties are very important to evaluate the potential usage of thermally treated products in the packaging sector, because they define gas and water vapour transfer. The experimental data prove that the thermal treatment reduces the value of mass diffusivity probably due to the chemical modification of the cell wall. However, the permeability value does not change significantly, probably because the thermal treatment has a slight impact on the porous structure of wood.

Modifikation des Massentransports in Pappelholz durch Hitzebehandlung bei hoher Temperatur

Zusammenfassung Diese Arbeit behandelt den Massentransport im hygroskopischen Bereich von Pappelholz vor und nach der Behandlung bei 200°C. Zwei Eigenschaften werden an den gleichen Proben bestimmt: zum einen die Diffusionseigenschaft (Massenfluss unter dem Einfluss eines Konzentrationsgradienten), zum andern die Gasdurchlässigkeit (Massenfluss unter dem Einfluss eines Druckgradienten). Diese beiden Eigenschaften sind wichtige Kenngrößen für die mögliche Verwendung der Produkte im Verpackungsbereich, da sie die Bewegung von Gas und Wasserdampf definieren. Die experimentel-

len Daten zeigen, dass die thermische Behandlung, wahrscheinlich aufgrund chemischer Veränderungen der Zellwand, den Massenfluss reduziert. Dagegen ändert sich die Gasdurchlässigkeit nicht signifikant, da die thermische Behandlung nur einen geringen Einfluss auf die Porenstruktur des Holzes ausübt.

1 Introduction

Torrefaction is a physical treatment for wood that does not require external chemical products. The main transport phenomenon is the thermal flux that induces both physical and chemical modifications inside the wood. This process seeks to control the pyrolysis to exploit the thermal degradation of the wood constituents without oxygen to avoid oxidation. The pyrolysis is carried out between 200°C and 260°C, giving the wood new physical, chemical and mechanical properties. Although this process sounds simple there are numerous parameters that control this process because of the complexity of the wood. The literature on this moderate heating process indicates that it has been studied for some time (Stamm 1946; Buro 1954; Kollmann and Topf 1971; Burmester 1973; Bourgois and Guyonnet 1988; Bourgois et al. 1989; Ruyter 1989). Recent efforts on thermal treatment of wood have lead to the development of several treatment processes previously or presently introduced to the European market, especially in Finland, France and some other European countries (BRE-CT-5006 1998). The various wood improvement processes are documented in some patent specifications ((EP0018446 1982; EP0612595 1994; EP0623433 1994; ARMINES 1995). More recently, a report (Rapp 2001) provides useful information on each of the four different major European heat treatment of wood thermally modified after these processes:

- The Finnish Thermowood
- The Dutch Plato wood
- The French Retification
- The German Oil heat treatment

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Heat treatment of wood has an effect on the chemical composition of wood. In most of the publications on the heat treatment of wood reference is made to improved dimensional stability and increased resistance to fungi, though also to negative changes in some other wood characteristics. The chemical degradation of wood occurs in the hemicellulose, cellulose and lignin components. The changed wood composition results in a lower hygroscopicity with a major influence on both dimensional stability and durability. The higher the treatment temperature the better the durability, stability and biological properties of the product, however, the overall mechanical properties of the wood are weakened. In order to reach a selective degree of depolymerisation of the hemicellulose during the treatment, relatively mild conditions can be applied to limit unwanted side reactions, which can influence the mechanical properties negatively (Tjeerdsma et al. 1998). In the numerous papers devoted to this process, interest is focused often on the mechanical characteristics as well as increased dimensional stability and durability, but never on the mass transfer properties.

The objective of this study is to determine the effects of a thermal treatment on two mass transfer properties of Poplar wood:

- the mass diffusivity, which defines the mass flux in response to a concentration gradient,
- the air permeability, which defines the mass flux in response to a pressure gradient.

In order to be sure to draw clear conclusions on the effect on the thermal treatment, measurements have been carried out on the same samples, before and after the treatment at 200°C. In addition, duplicate samples have been prepared on the same board, i.e. along the same longitudinal line and at different radial positions within the log. The analysis of the experimental parameters allows information to be obtained on the morphology of the porous medium before and after thermal treatment. To our knowledge this work is the first one proposed on this topic.

2 Material and methods

2.1 Wood sampling

The samples used for testing come from poplar logs supplied by SIEL (Syndicat Interprofessionnel des Emballages Légers). The logs have been cut in 10 mm thick planks and have been dried artificially at Cirad according to a schedule provided by CTBA (Centre Technique du Bois et de l'Ameublement). The stack has been stored in a conditioned chamber with psychrometric conditions of 60% relative humidity and 20°C dry bulb. Before heating, each sample had an average moisture content of 12%. Five round samples have been cut respectively from planks cut in the heartwood zone, intermediate zone and sapwood zone using a special cutting instrument with an internal diameter 72 mm (Fig. 1). After cutting, the lateral faces of the samples are insulated by applying two coats of an epoxy resin to avoid penetration and leakage of water vapor at the lateral face of the sample during the measurements. The experimental devices (both designed and developed at LERMAB) have been conceived to use the same

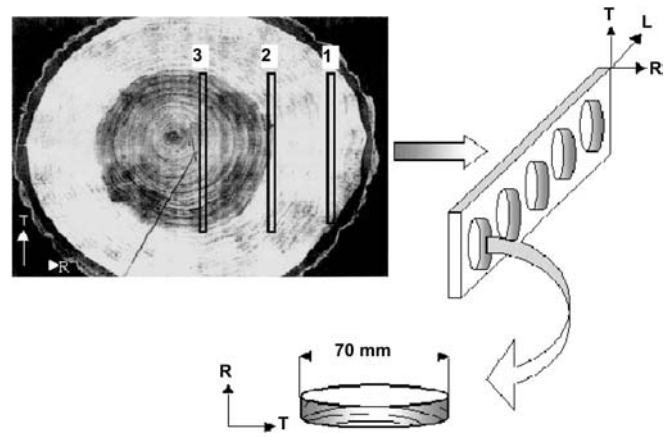


Fig. 1 Sampling: Five round samples have been extracted respectively from sapwood (1), intermediate (2) and heartwood (3) zones
Abb. 1 Probenahme: fünf Scheiben wurden jeweils von Splintholz (1), Übergangsholz (2) und Kernholz (3) entnommen

sample geometry (disks of 70 mm in diameter and between 5 and 20 mm in thickness). This means that both mass transport parameters, diffusivity and permeability, are determined on each sample, which allows the values to be analysed and compared with very good accuracy, in spite of wood variability.

2.2 Steady state determination of the mass diffusivity of wood in the hygroscopic domain

The moisture movement in the hygroscopic domain of wood is governed by complex phenomena that include binary molecular diffusion in the gaseous phase and migration of bound water in the cell walls. In addition, these phenomena interact at the microscopic scale, acting in series, or in parallel, according to the pore morphology. The macroscopic flux results from a coupling between these phenomena at the microscopic scale (Perré and Turner 2001). Nevertheless, in the case of isothermal and steady-state conditions, one knows that all possible formulations are equivalent (Siau 1984). We choose here the driving force that should be used for the binary diffusion of water-vapour in air

$$q = -\rho_g D_{eff} \nabla \left(\frac{\rho_v}{\rho_g} \right) \cong -f D_v \nabla (\rho_v) \quad (1)$$

where:

q Water-vapour flux through the specimen ($\text{kg.s}^{-1}.\text{m}^{-2}$),
 ρ_g gaseous density, kg.m^{-3}
 ρ_v water vapour density, kg.m^{-3}

and

$$D_{eff} = f D_v \quad (2)$$

where:

D_{eff} diffusion coefficient in gross wood, $\text{m}^2 \text{s}^{-1}$
 D_v diffusion coefficient of water-vapour in the bulk air

f = Dimensionless diffusivity

$$= \left[\frac{\text{coefficient of vapour diffusion in wood}}{\text{coefficient of vapor diffusion in air}} \right]$$

The dimensionless diffusivity f is the ratio between diffusion of water vapour in wood and diffusion of water-vapour in air. It accounts for the resistance to the water-vapour migration due to the

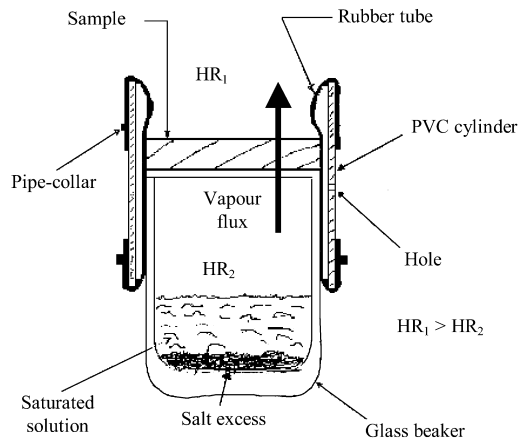


Fig. 2 Schematic of the device "PVC-CHA" used to determine the mass diffusivity of wood in the hygroscopic range
Abb. 2 Schema des Messgeräts "PVC-CHA" zum Bestimmen der Massen-Diffusivität im hygroskopischen Bereich

presence of wood compared to diffusion of vapour in the same layer of air. This parameter varies from 0 to 1 and thus gives an easy interpretation of the material behaviour (for an impermeable material $f=0$, and for a very diffusive material, such as galls wool, $f \approx 1$).

2.3 Experimental device

A detailed description of the material used in this part can be found elsewhere (Zohoun and Perré 1997; Zohoun 1998; Agoua and Zohoun 2001). Steady-state determination of the mass diffusivity has been chosen: two different values of the air humidity are applied on each side of the sample. The mass flux established in steady-state is then determined by weighing. As explained by the authors, this technique, which is slow but accurate and trouble-free, is now widely used in the LERMAB. The measurements might take several days, basically two weeks for a set of samples (10 to 20 devices can be placed in the same climatic chamber).

The ability to maintain a constant relative humidity over a long is a key requirement to determine accurately the mass diffusivity in steady-state regime. This is why we used a climatic chamber specifically conceived and built for this purpose. A box made of aluminium panels is separated in two parts by another horizontal panel: an upper volume where the specimens are placed and a lower volume where a tub containing distillate water. A ventilator generates an air circulation between the two partitions, through the two opposite apertures. The forced-air flows over the free surface of the water in the tub, maintained at the dew point temperature by a water-cooler. A dew point analyser has controlled the actual dew point temperature of the air. The Plexiglas door is equipped with two holes closed with diaphragms. These holes allow the sample to be weighted without perturbing either temperature or relative humidity.

The vapometer permits the air humidity to be imposed on the other side of the sample. It comprises a glass beaker containing a salt solution, the specimen placed on top and a pneumatic device named PVC-CHA system¹ (Fig. 2). The latter consists of a PVC pipe ($\varnothing=7$ cm, length=10 cm) covered inside with a piece of a rubber tube of smaller diameter than the PVC cylinder (hence the parabolic shape of the rubber tube). By using a vacuum pump, the air between the pipe and the rubber tube can be aspirated through a small hole in the pipe, causing the adhesion of the inner tube to the pipe, allowing the application of the pneumatic vice to the specimen and glass beaker. When the air is released, the pneumatic

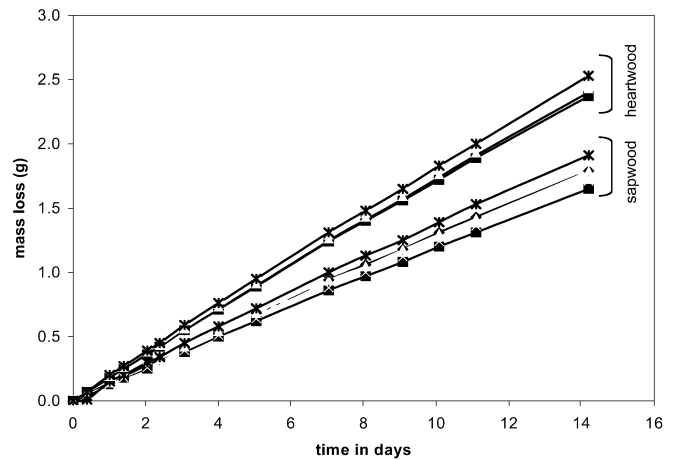


Fig. 3 Evolution of mass loss according to time for the 10 samples from sampling zones 1 and 3

Abb. 3 Verlauf der Massenverluste für 10 Proben aus den Bereichen 1 und 3

vice sticks strongly to the specimen, ensuring vapour tightness around the sample during the experiment. The improvement of this pneumatic device compared with others vapometers, is due to its elasticity and to its free adhesion to the specimen which minimises the vapour dispersions even when deformations (swelling or shrinkage) of the specimen shape occur during the test. In addition, it is very easy to insert or remove the sample.

2.4 Measurement protocol

Every specimen is placed on the top of a glass beaker containing a salt solution (NaCl) giving a RH value very close to 75%. Then the whole devices are inserted inside the climatic chamber at a RH of 40% and their weight is measured twice a day. The RH difference between the climate chamber and the glass equals 35% and the partial pressure difference is 1914 Pa. Only the linear zone of the mass loss is used to calculate the flux. Reduced diffusivity value f is calculated by the following formulae, derived from Eq. 2

$$f = \frac{Q}{D_v A} \times \frac{e}{(P_2 - P_1)} \times \frac{RT}{M_v} \quad (3)$$

Q mass flux measured (kg s^{-1})

A sample cross section (m^2)

M_v vapour molar weight (kg mole^{-1})

P_1 vapour partial pressure at a relative humidity of 40% in the climatic chamber

P_2 vapour partial pressure at a relative humidity of 75% inside the vapometer

T Testing temperature (K)

R constant of perfect gas

e sample thickness (m)

Figures 3 and 4 depict the mass loss curves obtained for each sample of series native 1 and 3 in one hand, and, in the other hand, the effect of thermal treatment on the samples of series 3. For untreated samples, it is important to note that diffusivity varies from heartwood to bark. Closed values obtained for repetitions inside the same plank (consequently for the same radial position in the log), reinforce this observation. Moreover, the difference of behaviour between two sampling zones remains significant after the thermal treatment.

¹ CHA: from "chambre à air" (inner tube in French)

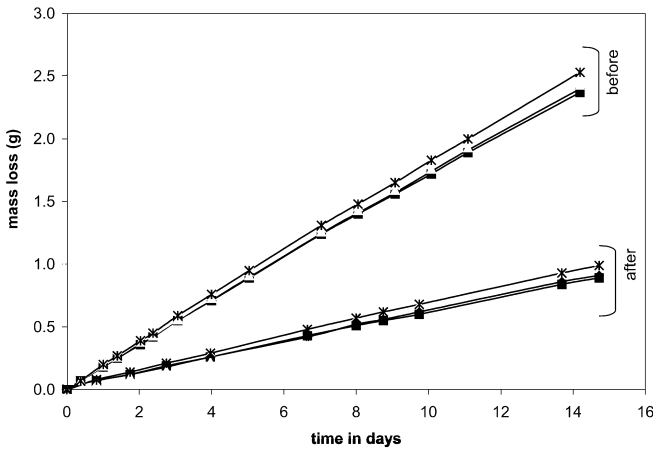


Fig. 4 Evolution of mass loss for the samples of the series 3 (heartwood) before and after thermal treatment

Abb. 4 Verlauf der Massenverluste für Proben der Serie 3 (Kernholz) vor und nach der Hitzebehandlung

2.5 Determination of wood permeability to air in the hygroscopic domain: Convective gas transfer in a porous medium

When a porous medium is permeable, a fluid flow can be generated when a pressure gradient exists within this medium. In the case of single-phase transport, a linear relation is often assumed to be valid between the fluid velocity and the pressure gradient. This relation is known as being Darcy's law:

$$\mathbf{v} = -\frac{K}{\mu} \nabla(P) \quad (4)$$

where:

- $\mathbf{v}$ apparent velocity of the fluid through the specimen (m.s^{-1}),
- K permeability, m^2
- μ dynamic viscosity of the fluid (Pa.s^{-1})

Although numerous deviations from this linear law have been reported in the literature (Siau 1984; Dullien 1992), its validity will be assumed throughout this work. Indeed, in configurations with usual pressure and pressure gradient, these deviations, due to recirculation, turbulent flow and slip flow (for a gaseous phase), remain of negligible effect.

2.6 Experimental device

The need to carry out measurements of permeability to air of various species, along the three material directions and at different pressure levels requires an accurate and reliable device capable of measurement over a wide range of permeability values (from 10^{-10} down to 10^{-18} m^2). For this purpose, Perré and Agoua (1999) developed a system called ALU-CHA at LERMAB. The originality of this medium lies in a high reliability of lateral airtightness, the rapid operation and the possibility to measure permeability with the same geometry, thus on the same samples than those employed for the diffusion measurement (Fig. 5). This device allows an accurate and constant pressure difference to be applied between the two faces of the sample. The corresponding gas flux is determined by the measurement of the thermal perturbation due to the air flux when it passes through a capillary tube.

The ALU-PCV system developed to handle the sample resembles the PVC-CHA system. The same principle is used to ensure the lateral air tightness. However, because the measured flux can be very low, absolutely no lateral leakage can be accepted. This is why the PVC cylinder has been changed for an aluminium cylinder. In

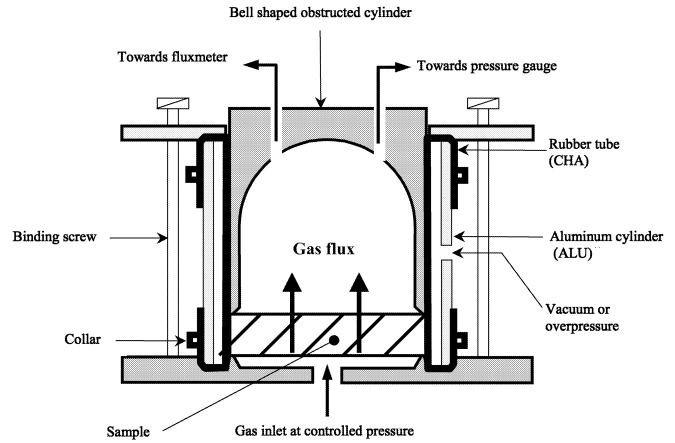


Fig. 5 Diagram of the sample support with ALU-CHA system

Abb. 5 Skizze der Probenauflage im ALU-CHA-System

addition to the vacuum phase used to remove the obstructed cylinder and place the sample, the chamber between the rubber tube and the aluminium tube is now strong enough to support an important overpressure. Typically, 2 to 3 bars are applied in this chamber during the test to press the rubber joint on the sample.

The experimental device is equipped with a small membrane pump and a 20-litres tank. This tank supplies the pressure controller with air as well as a Venturi system that produces the under-pressure needed to handle freely the obstructed cylinder and the sample. Once all pieces are correctly placed, the partial vacuum level is changed for the overpressure that exists in the tank. This overpressure ensures airtightness both around the sample and around the obstructed cylinder. Then, the operator has to choose the pressure gap across the sample, so that both the pressure level and the gas flux are accurately determined. Several pressure gaps and corresponding fluxes are collected at steady-state, so that the linear relationship can be checked.

Take into consideration the compressibility of the air, the permeability value, K , is calculated from Darcy's law (Eq. 4) using the following expression (Perré 1987):

$$K = \frac{Q \cdot \mu \cdot e \cdot P}{A \cdot \Delta P \cdot \bar{P}} \quad (5)$$

with:

- K Intrinsic permeability (m^2)
- Q Volumetric flux (m^3/s)
- μ Dynamic viscosity of the air (Pa.s)
- e Sample thickness (m)
- P Pressure at which flux Q is measured (Pa)
- A Area of the right section of the sample (m^2)
- ΔP $P_2 - P_1$ = pressure gap (Pa)
- $\bar{P}$ $(P_1 + P_2)/2$ = averaged pressure (Pa)

For the dynamic viscosity of humid air, a numerical value of $1.97 \cdot 10^{-5} \text{ Pa.s}$ was used in the calculations (Kestin, Whitelaw 1963).

3 The thermal treatment

The experimental device (Fig. 6) is composed of an oven with an electrical resistance regulated in temperature, an O_2 and CO_2/CO analyser and an ordinate analogue to digital converter (ADC) linked to a computer in order to control the operating conditions. A fan forces the air to circulate in the chamber in a closed loop.

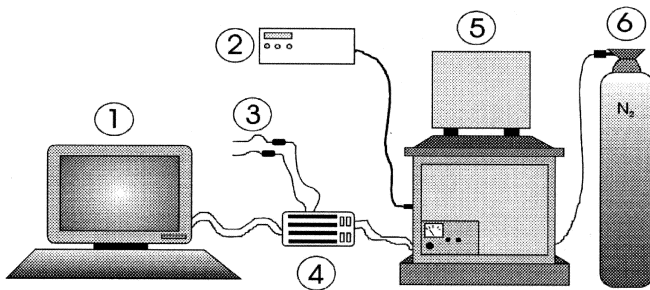


Fig. 6 The experimental device used to proceed to thermal treatment with: 1—monitoring; 2—O₂ analyser; 3—probes; 4—data capture; 5—reactor; 6—nitrogen

Abb. 6 Versuchsanordnung zum Verfolgen der Hitzebehandlung: 1—Beobachten; 2—O₂-Analysator; 3—Sonden; 4—Datenaufnahme; 5—Reaktor; 6—Stickstoffzufuhr

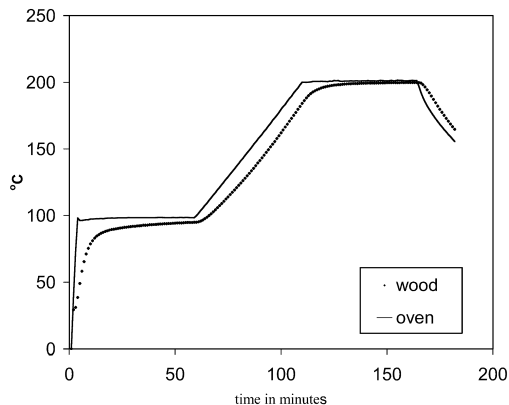


Fig. 7 Evolution of the temperatures in the oven and in the wood
Abb. 7 Temperaturverlauf im Ofen und im Holz

Once all measurement have been done on the natural samples, the following operating conditions have been applied:

- 1- Temperature rise up to 150°C, heating speed 2°C /min
- 2- Drying stage at 150°C during 1 hour
- 3- Temperature rise up to 200°C, heating speed 1°C /min
- 4- Stage at the final temperature 200°C, 1 hour
- 5- Natural cooling

Different operating conditions were tested to assess the excellent reliability of the regulation system. Figure 7 presents the temperature values collected versus time in the oven and in different positions within the sample. Using a copper sample of similar geometry, we also determined the average thermal exchange coefficient and found $H_f=28 \text{ W/m}^2/\text{°C}$

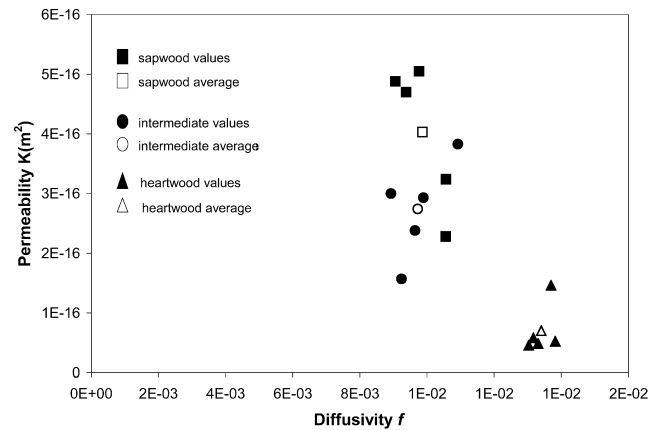


Fig. 8 Permeability vs. diffusivity of untreated wood samples
Abb. 8 Beziehung zwischen Permeabilität und Diffusivität für unbehandeltes Holz

4 Experimental results and discussion

4.1 Untreated wood

Figure 8 represents permeability and diffusivity measurements for each untreated wood sample. Table 1 gives the values measured for each sample as well as the centred mean and the standard deviation. These values depict significant variations according to the sampling zone in the log, from the outer zone, in sapwood to the inner zone, in heartwood. As expected, the heartwood samples are significantly less permeable than those coming from the sapwood zone (Ward 1986; Bao and Lu 1999). This trend is reinforced by the small variation observed within each zone. We can note that, on the three sets of samples, the permeability values vary by a factor 10 (0.5 to 5.10^{-16} m^2) for untreated wood samples. Such a variation along the radius is typical of permeability values measured on wood. The ratio can easily attains 100 for softwood (Comstock and Cote 1968; Siau 1984). Note also that the same samples depict an averaged diffusivity greater in sampling zones 1 and 2.

Samples from the outer part of the log (sampling zones 1 and 2) present a low diffusivity variability (values between 0.009 and 0.011) but a quite important scattering of the permeability values ($1.57.10^{-16}$ to $5.05.10^{-16}$). The scattering is reduced for the samples from the heartwood zone (values centred at 0.013 for f and $0.7 \cdot 10^{-16}$ for K).

4.2 Effect of thermal treatment on transfer properties

For all tested samples, the thermal treatment has reduced significantly the diffusivity value. After treatment, the values range from 0.006 to 0.009 (Fig. 9). Samples from sampling zone 3 (heartwood) seem more sensitive to the thermal treatment than samples from zones 1 and 2 (sapwood). Decrease of diffusivity for this zone is around 60% versus 40% for zones 1 and 2. Diffusivity is a wood

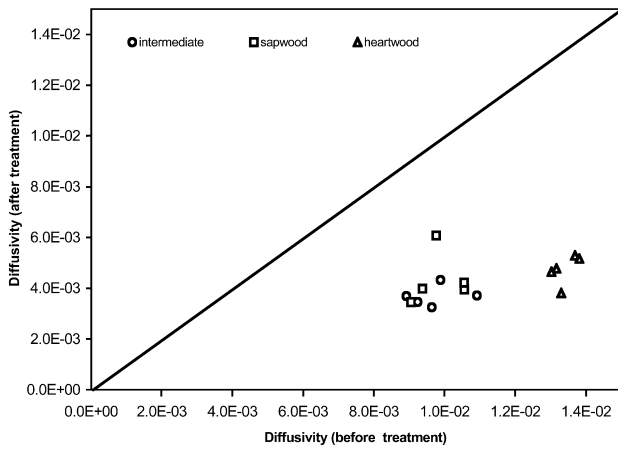


Fig. 9 Relation between reduced diffusivity f of treated wood and untreated wood in the radial direction
Abb. 9 Beziehung zwischen den reduzierten Diffusivitäten von behandeltem und unbehandeltem Holz in radialer Richtung

property resulting from its anatomical morphology and the composition of the cell walls. In order to understand the variability encountered in different species, authors like Siau (Siau 1984) involve the anatomical structure, others (Hillis and Rozna 1985a, b; Rice and D'onofrio 1996; Tjeerdsma et al. 1998) complete the explanation by the presence of different chemical elements. From a microscopic point of view, the solid phase constituting the cell wall is not inert and gives rise to bound water migration. The thermal treatment strongly affects its chemical composition. The destruction of some chemical constituents, especially hemicelluloses (Bilbao and Millera 1989) molecules the least thermally stable, provokes a decrease of the number of hydroxyls groups. This is the major explanation for the reduction of the hygroscopicity. But, we have to keep in mind that bound water diffusion is a mechanism where water molecules are able to jump from one site to the other (Skaar 1988). So, it is likely to expect than a reduction of hydroxyl groups is able to lower the ability of water molecule to migrate. This is certainly an explanation for the general tendency observed in our measurements.

Figure 10 depicts the values of untreated and treated wood permeability. It gives information on the evolution of this property due to the thermal treatment for the three radial positions. For the three treated lots, permeability values vary by a factor 7 (0.6 to $4 \cdot 10^{-16} \text{m}^2$). The quite low deviation observed for all positions within the log before and after treatment (all points remain close to the bisecting line) allows us to say that the treatment did not affect permeability. In particular, we can conclude that the thermal treatment did not provoke additional cracks inside the samples and that the porous structure should not change significantly. This observation gives also a indirect proof of the good quality of the drying operation.

Figure 11 depicts the representative points of each sampling zone (pondered mean) in the diffusivity-perme-

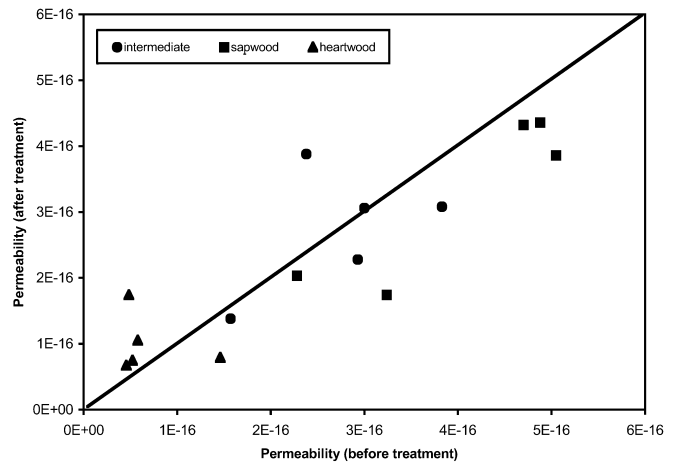


Fig. 10 Permeability of untreated and treated wood samples
Abb. 10 Permeabilität von unbehandelten und behandelten Proben

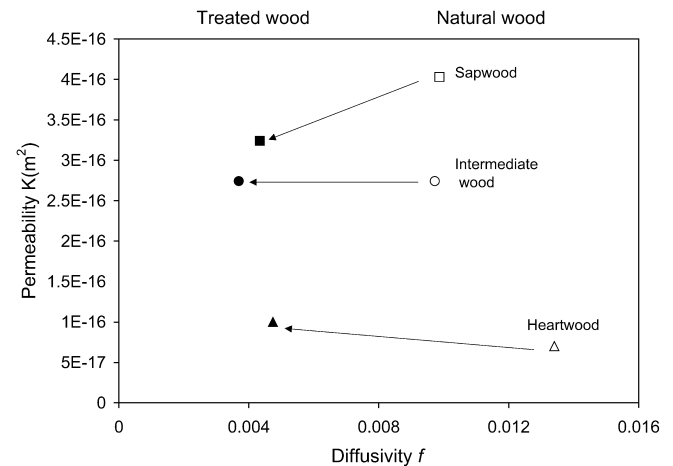


Fig. 11 Permeability versus diffusivity: untreated and treated wood samples
Abb. 11 Permeabilität in Abhängigkeit von der Diffusivität: unbehandelte und behandelte Proben

ability graph, before and after the thermal treatment. In particular, this figure points out the change of position due to the thermal treatment and summarises by itself the general tendencies previously observed. Whatever the sampling zone, the treatment has no, or few, effects on wood permeability (the arrows are almost horizontal) whereas diffusivity is significantly reduced.

5 Conclusion

Measurements of mass diffusivity and permeability have been determined on Poplar samples to investigate the effect of thermal treatment on these physical properties. Two specific experimental devices have been used that employ the same sample geometry. This allowed both properties to be determined on each sample, before and after the thermal treatment. Duplicate samples have been

selected and prepared in three different zones of the log (outer zone, intermediate zone and inner zone).

As expected, the experimental data depict a significant variation along the radial position (from sapwood to heartwood). In addition, we observed that, for each zone, the thermal treatment process has decreased the magnitude of mass diffusivity, without affecting permeability. The evolution of these parameters highlights that the thermal treatment does not affect the pore morphology and does not promote cracking provided a good drying process has been applied previously. On the other hand, the evolution of the chemical composition of the cell wall, namely the degradation of some hydrophilic components (hemicelluloses) reduces significantly the ability of bound water to migrate through the wood sample.

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